

UNIVERSITY OF WASHINGTON
Department of
CHEMISTRY AND CHEMICAL ENGINEERING
SEATTLE

May 8, 1942

Dear Mr. Cheyne,

As yet we have not received payment on the vouchers covering the phenol analysis. Is this delay normal or do you suppose they have been redetracked someplace?

Sincerely,
R. W. Moulton

P.S.

Your letter of the 25th came.



UNIVERSITY OF WASHINGTON
Department of
CHEMISTRY AND CHEMICAL ENGINEERING
SEATTLE

March 31, 1942

Mr. H. Cheyne
State Pollution Laboratory
Gig Harbor, Washington

Dear Mr. Cheyne:

Your letter of March 28 has been received. I mailed you the results of the analytical work yesterday together with a statement for \$25.00 for the work done.

I have signed the state vouchers and had them notarized as requested. They are enclosed with this letter.

In answer to your questions:

First, the concentration is in parts per million by weight. The difference between the two titrations (blank and sample) multiplied by the factor 0.0015675, for 0.1 N sodium thiosulphate, gives the weight of phenol and cresols in terms of phenol. This weight multiplied by one million and divided by the weight of the sample will give parts per million by weight. If the normality of the sodium thiosulphate is different than 0.1 N insert an appropriate correction factor in the numerator to take care of that fact.

Secondly, the size of the aliquot taken to contain about 0.02 gm. of phenol can only be estimated if you do not have any previous analysis on the sample. After running one sample if you find the size of aliquot taken is not close to the desired value a second sample should be run with a correction made in the size of sample taken.

I hope that this answers your questions and that I have filled out the vouchers correctly.

Very truly yours,

R. W. Moulton

R. W. Moulton
Asst. Prof. in Chem. Eng.

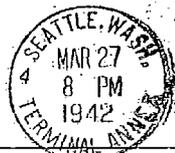
RWM:cn
Enclosure

3-27-42

Dear Mr. Chayne,

We have completed your samples and I will try and get out the complete report tomorrow. The figure I gave you yesterday on the first sample you brought in should be multiplied by 10 is about 50 ppm after your caution. Runs on phenol. No mixtures check method OK.

Sincerely,
R. W. Mante



THIS SIDE OF CARD IS FOR ADDRESS

H. CHEYNE
WASH. STATE POLLUTION BOARD
GIG HARBOR, WASHINGTON

Phenol Analysis of Sand

Samples taken April 2, 1942 ~~at outlet on~~
Beach near outlet to Western Gas Co. waste outlet

Sample No.	Location	ppm Phenols.							
1A	2' tide level over pipe	22.66							
2A	3' tide level over pipe	—							
3A	4' tide level over pipe	—							
4A	5' tide level over pipe	50.79							
5A	6' tide level over pipe	70.22							

PHENOL DETERMINATION

Purification

Reagents: 0.1 N bromine (2.784 gr. potassium bromate, 10 gr. potassium bromide per liter). 0.1 sodium thiosulphate (24.82 gr. per liter). Con. HCl, pulverized crystals of potassium iodide, starch sol., 20% sodium hydroxide sol., powdered lead carbonate, C.P. sodium bicarbonate, 30-mesh sand, and castor oil.

Procedure: Measure 25 cc. into a 350 cc. Kjeldahl flask, add 5 cc. of 20% NaOH and 25 cc. of water. Boil off ammonia until vapors have no effect on moist red litmas paper. Add 1 gr. lead carbonate and boil 1 minute longer. Weigh out 15 gr. of sodium bicarbonate and add to contents of flask. Make up volume to 75 cc., add a few grains of sand to prevent bumping, a drop of castor oil to stop frothing, and distill 50 cc. of it. Repeat twice with addition of 25 cc. distilled water each time. Cool the receiving flask under tap or in a trough.

75 cc
Treatment: Take an aliquot estimated to contain about 0.02 grams of phenol or cresol in terms of phenol and transfer to a 500 cc. glass stoppered flask. Add just enough distilled water to make volume 200 cc. Add 25 cc. (measured) of potassium bromide sol. containing 250 grams in one liter. Add 10 cc. of 1:1 HCl sol. and place in a water bath held at 25°C. Let stand until tem of sol. is 25°C. Add exactly 25 cc. of potassium bromate sol. containing 3 grams in one liter. Mix well, then let stand at 25°C. (preferably with in one degree, at most with in two degrees) for exactly 60 minutes, shaking two or three times during this period. At the end of this time add 25 cc. of potassium iodide sol. containing 200 grams in one liter, mix well, and let stand at 25°C. for exactly 30 minutes, shaking two of three times during this period.

213.1 Bb. A
212.5
43
Titrate the liberated iodine with 0.1 N. sodium thiosulfate, adding starch sol. near the end of the titration and disregarding any return of the blue starch-iodine color. Run a blank determination in the same manner.

0.94 g?
The difference between the two titrations times 0.0015675 equals the weight of phenols and cresols in terms of phenols. The calculation is made according to the empirical reaction for cresols.

References:

Scott, R. D. 1939 Application of a Bromine method in determination of phenol and cresol. Ann. Ed. Ind. Eng. Chem. # 31, Jan. 15, 1931: 67-70.

Williams, Ralph D. 1927 Rapid determination of phenol in ammonia liquor liquor and other solutions. Ind. Eng. Chem. 19, 4. April 1927: 530-531.

Reagents for Purification

20% Sodium hydroxide (200 cc - 18.75 N) ✓

Lead carbonate

Sodium bicarbonate

Sand

Castor oil

Reagents after Purification

Potassium bromide (250 grs/l) (2L) ✓

Hydrochloric acid (1:1 sol) (200 cc) ✓

Potassium bromate (3 gr/l) (2L) ✓

Potassium iodide (200 gr/l) (2L) ✓

Sodium thiosulfate 0.1 N (24.82 gr/l) (2L)

Starch

-Tests of
 Test on ciliary Mechanism of
 Pacific Oyster when subjected to 5 P.P.M.
 of Phenols in Gas Plant liquor.

Test Conducted on March 23, 1942

This test determines the length of time required for an oyster to pump a given amount of water through a tube for a distance of 15 centimeters. The oyster first tested in unpolluted sea water then in water containing 5 P.P.M. phenols in Gas Plant liquor. Trials were made at different intervals.

Results.	5 P.P.M. phenols immediately.	5 mi.	15 mi.	30 mi.
Sea water 4.63 sec.	6.06 sec	5.17 sec	5.59 sec	8.09 sec.

Carmin Red was used as the color to form the cone.

Method used and described by Paul S. Galtsoff
 of Fish & Wildlife Service

Effects of Pulp Mill Poll. on Oysters.

Bull. U.S.B.F. #6 1931: 162-167

Science, 1926 Vol. LXIV : 233-234

Jour. Gen. Physiology, 1928, Vol. XI : 415-431

Bull. U.S.B.F., 1928, Vol. XLIV : 1-39.

Test of effects of 5 ppm
Phenols on the Pacific Oyster

Time tested	Control	0	5 mi.	15 mi.	30 mi.
	4.8	6.9	5.9	5.6	6.9
	4.7	6.7	4.9	5.4	7.5
	4.7	5.4	5.3	5.4	7.8
	4.8	6.9	4.8	5.4	7.8
	4.5	5.4	5.1	5.7	7.8
	4.8	5.1	5.1	5.9	8.4
	4.4	6.7	4.9	5.7	8.8
	4.4	6.3	5.2	5.7	9.8
	4.6	5.4	5.5	5.5	8.7
	4.6	5.2	5.0	5.6	7.4
Σ	46.3	60.6	51.7	55.9	80.9
M	4.63	6.06	5.17	5.59	8.09

waste Gas Plant liquor used in this test.
liquor contained 50 ppm phenols.

Calculation of amount of solids
settling on Beach in 10 yrs.

Flow per minute

15 gal

60 gts.

56.782 liters

Flow per hour

3406.920 liters

Flow per day.

81766.080 liters

Specific gravity

1.0012

Grams of solids waste per day. 81,864,199.2960 grams.

Total solids

6.69 p.p.m.

Approx. grams of waste per day.

82 million

Grams of solids deposited/24 hrs

548.58 grams.

Days per year

365 days.

Grams per year

200,231.70 grams

Grams per 10 years.

2,002,317.00

Grams per pound

454 grams.

Pounds of solids deposited in 10 years.

4410 pounds.

or 2.2 Tons.

15 gal / minute

$$\frac{4}{60} \text{ gal / minute} = 56.782 \text{ l / minute}$$

$$\frac{3406920}{60} \text{ l / hr.}$$

$$\frac{13627680}{24} = 568236.8 \text{ l / day}$$

$$81766080 \text{ cc / day}$$

$$\frac{81766080}{1.0012} = 81664199.296 \text{ cc / day}$$

6.69 gv / 1,000,000 gv.

$$\frac{6.69}{92} = 0.0727173913$$

$$\frac{548.58 \text{ gv deposited / 24 hrs.}}{365 \text{ days / year}} = 1.499945205$$

$$\frac{274290}{329148} = 0.833297$$

$$\frac{164594}{200231.70 \text{ gv / year}}$$

$$\frac{200231.70 \text{ gv / year}}{10 \text{ years}} = 20023.17 \text{ gv / 10 yrs.}$$

$$\frac{200231.70}{4410} = 45.401746$$

$$\begin{array}{r} 454 \overline{) 2002317} \\ \underline{1816} \\ 1863 \\ \underline{1816} \\ 471 \\ \underline{454} \\ 177 \end{array}$$

$$\frac{2002317}{454 \text{ gv / lb.}} = 4410 \text{ lbs / 10 years deposited on beach.}$$

64.5194
64.4806

.0388

62.6300
62.5844

.0456

65.9575
65.9090

.0485

69.6700
69.6355

.0345

1,001.6
 50

50050

50,05 | 1,000.7.752
 03.8800
 35.035

 .37650
 35035

 26150
 25025

 11250

1,001.2
 50

500600

50,06 | 1,000.919
 04.5600
 45.054

 5460
 5006

 4540

1,001.1
 50

50,0550

50,055 | 1,000.9.69
 048.5000
 450495

 345050
 306330

 447200

~~1,001.2~~

5006 | 1,000.6.69
 034500
 30036

 44640
 40048

 45920

Phenols (Sand)

3-10-42

Sample No.	Vol. of sand	cc. of water	cc. of extract	cc. of extract sample	ppm phenols in sample	pH				
8	380	1000	775	50	Trace	7.7	no life in mud samples.			
9	380	1000	910	50	Trace	7.7				
10	380	1000	915	50	Blank	7.7				
11	380	1000	980	50	Blank	7.7				
12	380	1000	765	50	Trace	7.7				
Control						7.9				

Phenols (Sand)

3-4-42

Sample No.	Vol. of Sand.	cc of water	cc of Extract	cc of Extract Sample	P.P.M. Phenols in sample	
30 59	1	380	1000	965	50	0.0
87	2	380	1000	940	50	0.0
91	3	380	1000	955	50	0.0
93	4	380	1000	940	50	0.0
98	5	380	1000	920	50	1.0
136	6	380	1000	850	50	1.0
144	7	380	1000	935	50	0.5
148						
149						
155						
151						

UNIVERSITY OF WASHINGTON
Department of
CHEMISTRY AND CHEMICAL ENGINEERING
SEATTLE

April 23, 1942

Dear Mr. Cheyne,

As yet we have not heard
anything relative to the work
done on the gas plant waste
samples. How is the investigation
working out? Were the
vouchers made out correctly?

Yours very truly,
R. W. Mautler

Location	Plant P/Day	Total Solids
Intake of Settling Tank	70.0	7.75
Clarifier	50.5	9.19
Upper Man hole	49.5 →	6.69
Lower Man hole	14.5	4.23
Outlet on beach	< 1.0	
Outlet from oil barrels		15.91

Sand sample 1 A

Date started 4-3-42

50 gr. of sand added.

5 cc of NaOH

25 cc of Distilled H₂O

1 gr PbCO₃

15 gr NaHCO₃ (sodium bicarbonate)

Diluted to 100cc instead of 75 cc

57 cc distilled

25 cc H₂O added and distilled

78 cc distilled

25 cc H₂O added and distilled

100 cc distilled

Sample saved

75 cc added of sample

125 cc Distilled H₂O

25 cc potassium bromide added

10 cc HCl

Put in water bath until 250 c

25 cc potassium bromate added

put in water bath for 1 hr.

25 cc potassium iodide

Put in water bath 30 mi.

Titrated with Na₂S₂O₄

285 cc of sample
50 cc taken to titrate

32.42 cc 150 cc x 5.7 = 213.29 cc Na₂S₂O₄

Control

200 cc Distilled H₂O

25 cc potassium bromide added

10 cc HCl

214.40

214.40 cc Na₂S₂O₄

x factor = 205.18 cc Na₂S₂O₄

÷ 2 = 102.59

Sand Sample 2A

Date started 4-3-42

50 gr. of sand added

5 cc of NaOH

25 cc of Distilled H₂O.

1 gr PbCO₃

15 gr NaHCO₃

Diluted to 100 cc

60 cc distilled

25 cc added & distilled

87 cc distilled

25 cc added & distilled

100 cc distilled

Sample saved

75 cc added from sample

125 cc distilled H₂O added

25 cc potassium bromide added

10 cc HCl

Put in water bath until 25° C.

25 cc potassium bromate added

Put in water bath for 1 hr

25 cc potassium iodide added

Put in water bath for 30 mi.

Titration

285 cc of sample

50 cc taken for titration

$$37.43 \text{ cc} \times 5.7 = 213.25 \text{ cc } \text{Na}_2\text{S}_2\text{O}_3$$

$$\text{X factor} = 204.08 \text{ cc } \text{Na}_2\text{S}_2\text{O}_3 \div 2 = 102.04$$

Control

200 cc Distilled H₂O added

25 cc potassium bromide added

10 cc HCl

285 cc of sample

50 cc taken for titration

$$37.28 \text{ cc} \times 5.7 = 212.50 \text{ cc } \text{Na}_2\text{S}_2\text{O}_3$$

$$\text{X factor} = 203.36 \text{ cc } \text{Na}_2\text{S}_2\text{O}_3 \div 2 = 101.68$$

Sand Sample 3A

Date started 4-3-42

50 gr. sand added

5 cc NaOH

25 cc distilled H₂O

1 gr. PbCO₃

15 gr. NaHCO₃

75 cc distilled H₂O added

53 cc distilled

25 cc distilled H₂O added

77 cc distilled

25 cc distilled H₂O added

140 cc distilled

Sample saved.

50 cc added of sample

150 cc of distilled H₂O

25 cc potassium bromide added

10 cc HCl

Put in water bath until + 50 c.

25 cc potassium bromate added

put in water bath for 1 hr.

25 cc Potassium iodide added

Put in water bath for 30 mi.

Titration

285 cc in sample

50 cc taken

$$37.63 \text{ cc } Na_2S_2O_3 \times 5.7 = 214.49 \text{ cc } Na_2S_2O_3$$

$$\times \text{factor} = 205.27 \text{ cc } Na_2S_2O_3 \div 2 = 102.63 \quad (223.15) \left(\frac{293.10}{273.15} \right) (8) = 212.67 \text{ cc } Na_2S_2O_3$$

293 cc in sample

50 cc taken

$$37.55 \text{ cc } Na_2S_2O_3 \times 5.86 = 223.15 \text{ cc } Na_2S_2O_3$$

$$\times \text{factor} = 203.53 \text{ cc } Na_2S_2O_3 \div 2 = 101.76$$

Control

200 cc added of distilled H₂O

25 cc potassium bromide added

10 cc HCl

San. Sample 4A.

Date Started 4-3-42

50 gr of sand added

5 cc of NaOH

25 cc of distilled H₂O

1 gr PbCO₃ added

15 gr NaBCO₃ added

75 cc distilled H₂O

52 cc Distilled

25 cc Distilled H₂O added

78 cc Distilled

25 cc Distilled H₂O added

100 cc Distilled

Sample saved

50 cc added of sample

150 cc distilled H₂O added

25 cc potassium bromide added

10 cc HCl

Put in water bath until 25° C.

25 cc potassium bromate added

Put in water bath for 1 hr.

25 cc Potassium iodide added

Put in water bath for 30 mi.

Titration

285 cc of sample

50 cc taken

$$37.80 \text{ cc Na}_2\text{SO}_3 \times 5.7 = 215.46 \text{ cc Na}_2\text{SO}_3$$

$$\times \text{factor} = 206.20 \text{ cc Na}_2\text{SO}_3 \div 2 = 103.10$$

Control

200 cc distilled H₂O add.

25 cc potassium bromide

10 cc HCl

285 cc of sample

50 cc taken

38.10

$$38.10 \text{ cc Na}_2\text{SO}_3 \times 5.7 = 217.17 \text{ cc Na}_2\text{SO}_3$$

$$\times \text{factor} = 207.83 \text{ cc Na}_2\text{SO}_3 \div 2 = 103.91$$

Sand Sample 5A

Date started 4-4-42

50 gr sand added

5 cc NaOH

25 cc Distilled H₂O

1 gr PbCO₃

15 gr NaHCO₃

75 cc Distilled H₂O

54 cc Distilled

25 cc Distilled H₂O added

75 cc Distilled

25 cc Distilled H₂O added

100 cc Distilled

Sample saved

Control

50 cc added of sample
150 cc distilled H₂O added
25 cc potassium bromide added
10 cc HCl

200 cc distilled H₂O added
25 cc potassium bromide added
10 cc HCl

Put in water bath until 250° C

25 cc potassium bromate added

Put in water bath for 1 hr.

25 cc potassium iodide added

Put in water bath for 30 min.

Titration

285 cc in sample
50 cc taken

37.69 cc Na₂S₂O₄ × 5.7 = 214.83 cc Na₂S₂O₄

× factor = 205.59 cc Na₂S₂O₄ ÷ 2 = 102.79

285 cc in sample
50 cc taken

38.10 cc Na₂S₂O₄ × 5.7 = 217.17 cc Na₂S₂O₄

× factor = 207.83 cc Na₂S₂O₄
÷ 2 = 103.91

Sodium Thio sulphate standardization

41.10

41.78 }
41.82 } 41.80

$$\frac{40.00}{41.80} = 0.957 \text{ factor}$$

	<u>Sample</u>	<u>Blank</u>	<u>Difference</u>	<u>X 0.0015675</u>
2	1A 102.06	102.59	0.53	0.000830775
3	2A 102.04	103.66	1.62	0.00255675
4	3A 102.63	101.76	0.87	0.00136675
5	4A 103.10	103.91	0.81	0.001269675
6	5A 102.79	103.91	1.12	0.0017556 37.4

X 1000000
Sample wt.

1A	22.16 ppm
2A	_____
3A	_____
4A	50.79 ppm
5A	70.22 ppm

371W
5 1.000

Specific Gravity and
Total Solids of Gas Plant wastes
Taken from Western Gas Co. on April 2, 1942

Location	Specific Gravity	Total Solids p.p.m.
Intake to settling Sump.	1.0010	7.75
Intake to cooler	1.0012	9.19
Outlet from cooler	1.0011	9.69
Intake to upper man hole	1.0012	6.69
lower man hole	Sample taken February 26, 1942	
	1.0020	4.23
Outlet from oil barrels	Sample taken March 7, 1942	
	1.0009	15.91

Specific Gravity + Total Solids
 cm. 4-2-42

Bottle No. 383

Outlet from cooler.

wt. of pycnometer + waste from gas plant.		40.2932
wt. of gas plant waste		24.9018
Specific gravity		1.0011
wt. of Crucible (#3)		65.4090
amt. of Gas plant liquor added		50 cc
wt. of Crucible after evaporation		65.9575
wt. of solids		0.0485
Total Solids	$\frac{0.0485}{(50)(1.0011)}$	<u>9.69 ppm</u>

Bottle No. 441

Inlet to upper manhole.

wt. of pycnometer + waste from gas plant		40.2953
wt. of gas plant waste		24.9039
Specific gravity.		1.0012
wt. of Crucible (#4)		69.0355
amt. of Gas plant liquor added		50 cc
wt. of Crucible after evaporation		69.0700
wt. of solids		0.0345
Total Solids	$\frac{0.0345}{(50)(1.0012)}$	<u>6.69 ppm</u>

Specific Gravity & Total Solids
of Gas Plant Wastes, Bremerton, Wash.

4-2-42

Bottle No. 178

Intake to settling sump.

wt. of pycnometer		15.3914
wt. " " + distilled water		40.2649
wt. of water		24.9735
No. cc. or grams		24.9735

wt. of pycnometer + waste from gas plant		40.2904
wt. of gas plant waste		24.8970
Specific gravity.		<u>1.0010</u>

wt. of crucible (#1)		64.4806
amt. of Gas Plant liquor added		50. cc
wt. of crucible after evaporation		64.5194
wt. of solids		0.0388
Total solids.	$\frac{0.0378}{(50)(1.0010)} =$	<u>7.75 ppm</u>

Bottle No. 168

Intake to cooler

wt. of pycnometer + waste from gas plant.		40.2946
wt. of gas plant waste		24.9032
Specific gravity		<u>1.0012</u>

wt. of crucible (#2)		62.5844
amt. of gas plant liquor added		50. cc
wt. of crucible after evaporation		62.6300
wt. of solids		0.0456
Total solids	$\frac{0.0456}{(50)(1.0012)} =$	<u>9.19 ppm</u>

Specific Gravity + Total Solids
of Gas Plant Wastes, Brenton, Wash.

3-7-42

Sample taken at Oil barrel outlet

Sample taken	3-4-42	at outlet of last oil drum.	
weight of pycnometer			15.3420
weight " " + distilled water			40.2867
wt. of water			24.9447
No. cc or grams			24.9447
weight of pycnometer + waste from gas plant			40.3101
weight of gas plant waste			24.9681

SP = 1.0009

Total Solids

	#1	#2	#3
wt. of Dish	65.9110	64.4842	62.5870
amt. of waste	100cc	100cc	100cc
wt. of Dish after evap.	66.1250	64.6125	62.7232
wt. of Solids	0.2140	0.1283	0.1362
avg. wt. of Solids	0.1595		

$$T.S. = \frac{(wt. of residue)}{(100)(SP \cdot gr.)} = \frac{0.1595}{(100)(1.0009)} = 1591 \text{ ppm}$$

PH. 8.60

Specific Gravity & Total Solids
of Gas Plant Wastes, Bremerton, Washington

2-26-42

Samples taken from lower man hole on Storm Sewer

wt. of Pycnometer	15.4172
wt. of " and distilled water	40.2467
wt. of water	24.8295
No. cc. of water	24.8295

weight of pycnometer & waste from gas plant	40.2977	SP = 1.002
weight of gas plant waste	24.8799	

Total Solids

	#1	#2	#3
wt. of dish	65.9101	64.4819	62.5856
amt. of waste	50 cc	50 cc	50 cc
wt. of dish after Evap.	65.9309	64.5041	62.6063
wt. of Solids	0.0208	0.0222	0.0207
avg. wt. of solids	0.0212		

$$T.S. = \frac{\text{wt. of residue}}{(50)(sp gr)} = \frac{0.0212}{(50)(1.0020)} = 4.23 \text{ ppm}$$

Plankton test

Make up Miquel Sol. (A + B)

Catch plankton from Busy Lagoon

Place some amount in several culture dishes containing Miquel Sol.

Count organisms present in each with Haemocytometer

Add phenol dilutions and W.G.P.L. keeping control.

Count organisms at 2 day intervals.

Oyster test

Study ciliary reaction to dilutions of phenols and to W.G.P.L.

Salmon Fry test

Determine mortality of fry under different concentrations of phenols and W.G.P.L.

Silver Salmon Fry test

Container of stock	Concn. in stock	cc/l	F.W. flour	Final conc.					
W.G.P.L.	2.5	27.7	10	2.2 ppm phenols	48600 ppm	W.G.P.L.	Killed 73.3% in 12		
W.G.P.L.	2.5	11.6	10	1.09 ppm phenols	21380 ppm	W.G.P.L.	0.0076 in 121		
Phenols	1	27.7	10	0.1 ppm phenols	(20 cc / 200 cc of 1000 ppm phenol sol.)				
Phenols	1	13.8	10	0.05 ppm phenols			no effect in 20 hours		
W.G.P.L.	2000	7.12	10	50 ppm W.G.P.L.	(40 cc of 50 ppm 1:2 fish food)				
Phenols	100	8.59	10	3 ppm Phenols	(200 phenol / 20 cc)				no effect in 48 hrs
Phenols	100	30.9	10	10 ppm phenols					